L3 ANSWER 1 OF 1 WPIX COPYRIGHT 2003 THOMSON DERWENT on STN
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TITLE: Epoxy resin moulding material prodn. - by adding boric acid (salts) to compsn. contg. epoxy resin, hardener and inorganic filler.
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PATENT ASSIGNEE(S): (TOKE) TOKYO SHIBAURA ELECTRIC CO
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PATENT INFORMATION:

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INT. PATENT CLASSIF.: C08K-003-38; C08L-063-00; H01L-023-30
BASIC ABSTRACT:
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Epoxy resin moulding material is prepd by adding (1) above 0.1 wt % (on the resin component) of at least 1 of boric acid and boric acid salts to a compsn contg (a)epoxy resin, (b) hardener; and (c)inorganic filler.

Component (a)is e.g. bisphenol A type epoxy resin, phenol novolak type epoxy resin, alicyclic epoxy resin. Pref (a) contains below 10 ppm of Cl ion and below 0.1 wt % hydrolysing chlorine. Component (b) is e.g. phenol novolak resin, cresol novolak resin. Component (c) is e.g. crystalline silica powder, quartz glass powder, talc, Ba sulphate powder. The amt of (c) used is 150-400 pts to 100 pts of (a) and (b). The amt of (1) (e.g. Na borate, Zn borate)used is 0.1-50 wt%.

The moulding material reduces or inhibits corrosion of metal electrode. It is suitable for the mfr of resin-sealed electronic parts such as semiconductor element.

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(54) Title of the Invention: Epoxy Resin Type Forming Material

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Description

1. Title of the Invention : Epoxy Resin Type Forming Material

2. Scope of the Pat nt Application

Epoxy resin type forming material characterized by the fact that at least one selected from the group of boric acid and salts of boric acid is added into the composition of which the essential components are epoxy resin, a curing agent for epoxy resin, and an inorganic filler, at 0.1 wt % or more of the above mentioned resin.

3. Detailed Explanation of the Invention

This invention relates to the epoxy resin type forming material, and especially it relates to the epoxy resin type forming material which is suitable for embedding the electronic parts such as the semi conductor elements, etc.

It is widely practiced to embed the electronic parts such as the transistor elements or integrated circuit elements, etc., by using a thermosetting resin type forming material, for example, an epoxy resin type forming material (compound) for low pressure molding. In this embedding which uses the resin type forming material, the embedding work is easier compared with the hermetic sealing method which uses a metal container or a ceramic container, etc., and this is an advantage, however, it has the inconvenience that it is less reliable for the moisture sealing. The following can be considered as the cause of the lack of reliability toward the moisture. First, the formed product made out of the epoxy resin type forming material has moisture absorbency, and in the case when the electronic parts are embedded, the moisture in the surrounding air penetrates the formed body layer, and it reaches to the surface of the electronic parts, and for example, the aluminum wiring, etc., becomes corroded and sometimes it causes breaking of the wires.

Secondly, the ionic impurities that were by-produced in the process of synthesizing the epoxy resin and that are remaining, become ionized by the action of the water that penetrates, and this helps to corrode the metal film, etc., which were included in the embedded electronic parts. Of course, the purification treatment is performed for the epoxy resin that is used for the embedding after it is synthesized, and an effort is made

to remove the above mentioned ionic impurities, etc., however, it is practically impossible to remove these completely.

Next, an example of these ionic impurities will be explained. For example, epoxy resin is produced by reacting a phenolic hydroxy group with epichlorohydrin in the presence of sodium hydroxide, however, this reaction also forms sodium ions, chloride ions or sodium chloride as the reaction by- products, and it is inevitable for these to be mixed into the epoxy resin as the ionic impurities.

In addition, although they are not ionic impurities, quite large amounts of unreacted material remains in the epoxy resin, where, the chlorine finally separates from the epichlorohydrin that reacted with phenolic hydroxy groups, and which is in the state of just before forming the epoxy ring but chlorine is still remaining without separating. This is called hydro separatable chlorine, and this exists at levels of about 10 to 1000 times the chlorine level of the ionic impurities. Hydro separatable chlorine differs from the chlorine of the ionic impurities, and it does not immediately leech out when water enters, however, it can be ionized by the action of heat or a catalyst, and it leeches out and this causes the metal electrodes, etc., to be corroded. In any case, in the case of the epoxy resin type forming material, the action of trace amounts of ions which are unavoidably mixed into the material, causes the corrosion of the electrode materials, etc., when it is used for embedding the electronic parts, etc., and it damages the function of the element, and this is inconvenient.

The inventors of this invention investigated considering the above mentioned situation, and as the result, they discovered that the corrosion of metal electrodes can be decreased or can be restricted when at least one of either boric acid or a salt of boric acid is added into the epoxy resin type forming material.

Therefore, this invention is to offer the epoxy resin type forming material which is

(suitable? *illegible*) to produce the resin embedded electronic parts with excellent reliability, based on the above mentioned discovery.

Next, this invention will be explained in detail. This invention is the epoxy resin type forming material characterized by the fact that at least one selected from boric acid and salts of boric acid is added into the composition of which the essential components are epoxy resin, a curing agent for epoxy resin, and an inorganic filler, at 0.1 wt % or more of the above mentioned resin.

The epoxy resins that can be used in this invention are those which have 2 epoxy groups in their molecule, and if it is used generally as the forming material, anything can be used, for example, bis phenol A type epoxy resin, phenol novolac type epoxy resin, cresol novolac type epoxy resin, or alicyclic epoxy resins, etc. This epoxy resin should be preferably such that the chlorine ion content is 10 ppm or less, and the content of hydrolytic chlorine is 0.1 wt % or less.

Those that can be listed as the curing agent for the epoxy resin, which is one component in the composition of this invention, can be the phenolic curing agents such as phenol novolac resin, cresol novolac resin, etc. The composition ratio of this curing agent should be selected in the way that the reaction of the epoxy group in the epoxy resin component and the functional group in the curing agent does not become unbalanced.

Those that can be listed as the inorganic filler which is another composition component in this invention, are for example, crystalline silica powder, quartz glass powder, talc, calcium silicate powder, calcium carbonate powder, barium sulfate powder, glass fibers, etc. Among these, crystalline silica powder or quartz glass powder is most preferred. The composition ratio of this inorganic filler varies depending on the type, etc., of the above mentioned resin that is selected (the epoxy resin and its curing agent), however,

it should be generally 150 to 400 parts per 100 parts of resin.

The following can be listed as the boric acid or borate that is specially added in this invention. First, ortho boric acid, meta boric acid, and boron oxide can be listed as the boric acids, and for example, ortho borate, meta borate and other various poly borates can be listed as the borates. To be concrete, these can be the salts of the alkali metals such as sodium or potassium, etc., the salts of the alkali earth metal such as magnesium, calcium or barium, etc., and the salt of metals such as zinc, manganese, cobalt, aluminum, etc., and ammonium salts, acidic borates such as manganese acid borate, etc., and the composite salts such as aluminum potassium borate, etc. These boric acids or borates may be used alone or in a mixed system of 2 or more, however, the amount of these to be added should be at least 0.1 wt % of the total sum of the resin components, namely, the epoxy resin and the curing agent for the epoxy resin. The reason for this is because the effect of adding (reduction or restriction of corrosion) does not appear if it is less than 0.1 wt %. Also, generally the effect is the same even if the content exceeds 50 wt %, so that the upper limit is about 50 wt %.

The forming material of this invention may be comprised of only the above mentioned epoxy resin, epoxy resin curing agent, inorganic filler and added components such as boric acid, etc., however, if necessary, a curing promotion agent, a mold separation agent, a coloring agent, a fire resisting agent, a silane coupling agent, etc., also can be added appropriately.

The above mentioned curing promotion agent can be for example, an imidazole compound or a tertiary amine. Natural wax, synthetic wax, metal salts of linear fatty acids, acid amides, esters or paraffins, etc., can be listed as the mold separation agents. Brominated epoxy resin, chlorinated paraffin, bromo toluene, hexa bromo benzene, antimony tri oxide, etc., can be listed as the fire retardant agents. Carbon black, etc., can be listed as the coloring agent.

The epoxy resin type forming material of this invention can be easily prepared when the raw materials that were selected in the prescribed composition ratio are sufficiently mixed in a mixer, etc., and thereafter, it can be melt mixed by a heat roll, and by applying the mixing treatment with a kneader, etc.

When the epoxy resin type forming material of this invention is used for example for embedding the semiconductor elements, etc., it gives high reliability. Namely, the occurrence of failures of the embedded semiconductor elements caused by breakage, etc., due to the corrosion of the aluminum electrodes can be greatly reduced or restricted even in the moisture resistance tests conducted in high temperature, high pressure steam, and the desired performance can be maintained over a long period of time. The reason for this effect can be considered to be as follows: the surface of the metal which constructs a part of electronic parts to be embedded, such as aluminum electrodes, etc., is made to be in the passive state by the action of the boric acid or borates, etc., that were specially added, and the electric field, and by this, the action of other ionic impurities such as sodium ions or chlorine ions is prevented or reduced.

Next, the action and effect of this invention will be explained referring to concrete examples.

Actual Example 1

The composition system was comprised of 170 weight parts of cresol novolac type epoxy resin of which the epoxy equivalent weight was 240, 20 weight parts of brominated epoxy novolac resin of which the epoxy equivalent weight was 290, 100 weight parts of phenolic novolac resin of which the molecular weight was 750, 700 weight parts of quartz glass powder, 10 weight parts of 2- undecyl imidazole, 5 weight parts of carnauba wax, 50 weight parts of antimony tri oxide, 3 weight parts of carbon black, and 3 weight parts of a silane coupling agent. And boric acid or borate were

selected in the amounts (weight parts) shown in Table 1, and these were put into the composition system, and they were heat - mixed by a roll, and then it was crushed, and thus, 8 types of forming materials were prepared, including the Comparison example.

Table 1

	Actual Example						Comparison Example1
	A	B	C	D	E	F	
Ortho boric acid	3	-	-	-	-	-	-
Boric acid tri oxide	-	3	-	-	-	-	-
Ammonium borate	-	-	4	-	-	-	-
Borax	-	-	-	2	-	-	-
Magnesium borate	-	-	-	-	5	-	-
Basic zinc borate	-	-	-	-	-	5? *illegible*	-
Pressure cooker test (failure occurring time hr)	120	130	120	110	100	90	20

Using the forming materials prepared in the above mentioned manner, the silicon element for evaluating the moisture resistance was trans- molded at the molding conditions of 170 °C for 3 minutes. 20 embedded elements which were obtained like this, were tested in the moisture resistance test by applying 10 V in high temperature, high pressure steam at 120 °C, or the PCT (pressure cooker test) was conducted, and the reliability was evaluated as the time when the breakage (occurrence of failure) occurred due to the corrosion of the aluminum wiring, and the results are shown together in Table 1.

Actual Example 2

The composition system was comprised of 200 weight parts of cresol novolac type epoxy resin of which the epoxy equivalent weight was 235, 100 weight parts of phenolic novolac resin of which the molecular weight was 750, 650 weight parts of crystalline silica powder, 50 weight parts of 2- phenyl imidazole, 6 weight parts of calcium stearate,